

Claims

1. A method for purifying (meth)acrylic acid
5 obtained by catalytic or redox oxidation, of a gas
substrate consisting of propane and/or propylene and/or
acrolein in the case of the fabrication of acrylic
acid, and of isobutane and/or isobutene and/or
tertbutyl alcohol and/or methacrolein in the case of
10 the fabrication of methacrylic acid, said gas mixture
mainly consisting of:
- propane and/or propylene or isobutane and/or
isobutene if previously contained by the
substrate;
 - 15 - final oxidation products;
 - the desired (meth)acrylic acid;
 - (meth) acrolein;
 - tertbutyl alcohol in the case of the fabrication
of methacrylic acid;
 - 20 - water vapour;
 - acetic acid with, in the case of the fabrication
of methacrylic acid, acrylic acid as a
byproduct; and
 - heavy products of side reactions,
- 25 according to which the reaction gas mixture is sent to
the bottom of an absorption column (C1) which is
supplied at the top and in countercurrent with at least
one heavy hydrophobic absorption solvent, the
absorption taking place in the presence of at least one
30 polymerization inhibitor to obtain:
- at the top of the column (C1) a gas stream consisting
of:
 - light incondensable compounds consisting of
propane and/or propylene or isobutane and/or
35 isobutene, according to whether acrylic acid or
methacrylic acid is fabricated and the products of
the final oxidation of the mixture;
 - major quantities of light condensable compounds
consisting of water and acetic acid in the case of

the fabrication of acrylic acid, or of water, acetic acid and acrylic acid in the case of the fabrication of methacrylic acid;

and

- 5 - (meth)acrolein;
- at the bottom of said column (C1), a stream consisting of:
 - (meth)acrylic acid;
 - the heavy absorption solvent or solvents;
 - 10 - the heavy products of side reactions;
 - the polymerization inhibitor or inhibitors; and
 - minor quantities of said light condensable compounds,

the stream issuing from the column (C1) is then sent to
15 a separation column (C2) in which a separation is carried out to obtain:

- at the top, a stream consisting of light impurities which are sent to the bottom part of the absorption column (C1); and
 - 20 - at the bottom, a stream consisting of:
 - (meth)acrylic acid in solution in the absorption solvent or solvents;
 - a small proportion of said light condensable compounds;
 - 25 - the heavy products of side reactions;
- and
- the polymerization inhibitor or inhibitors,

characterized in that the liquid stream (4) from the bottom of the column (C2) is sent as feed to the top of
30 a first separation section (S1) suitable for obtaining

- at the top, a gas stream; and
- at the bottom, a stream (9) essentially containing the absorption solvent or solvents stripped of the lighter compounds, said stream being recycled as feed
35 to the column (C1) directly or after removal of the heavy products contained in this stream;

said gas stream obtained at the top of the first section (S1), or the liquid stream generated by the condensation of this gas, being sent to the bottom of a

second separation section (S2) suitable for concentrating the intermediate heavy compounds of which the boiling point is between that of the solvent (or of the lowest boiling point solvent in the case of a solvent mixture) and that of (meth)acrylic acid, and suitable for obtaining:

- at the top, a gas stream; and
- at the bottom, a liquid stream that is sent to the top of the first section (S1),

10 said gas stream obtained at the top of the second section (S2), or the liquid stream generated by condensation of this gas, being sent to the bottom part of a third separation section (S3) suitable for obtaining:

- 15 - at the top, a gas stream which is condensed and partly recycled to the top of said section (S3), the remainder being tapped off as pure (meth)acrylic acid stripped of the heavy impurities; and
- at the bottom, a liquid stream which is sent to the top of the second section (S2).

2. The method as claimed in claim 1, characterized in that the sections (S1), (S2) and (S3) are the respectively bottom, intermediate and upper sections of the same column (C3), the stream (4) from the bottom of the column (C2) being sent to the column (C3) above the section (S1).

3. The method as claimed in claim 2, characterized in that the number of theoretical trays of the column (C3) is 8 to 25, particularly 10 to 20, the number of theoretical trays of each of the sections (S1), (S2) and (S3) of the column (C3) being respectively:

- 1 to 5, particularly 1 to 3;
- 1 to 10, particularly 1 to 5; and
- 3 to 20, particularly 5 to 15.

35 4. The method as claimed in either of claims 2 and 3, characterized in that the pressure at the top of the column (C3) is 2.7 to 27 kPa, particularly 6.7 to 24 kPa, the temperature of the bottom of the column (C3) is 150 to 250°C, particularly 180 to 230°C, and the

temperature of the top of said column (C3) is 40 to 110°C, particularly 65 to 95°C.

5. The method as claimed in one of claims 2 to 4, characterized in that the column (C3) is a distillation column provided with a bottom boiler, a top condenser, with a reflux rate T_R imposed at the top of 0.5/1 to 4/1, preferably of 0.5/1 to 2/1.

6. The method as claimed in claim 1, characterized in that the sections (S1) and (S2) are the respectively lower and upper sections of the same column (C3₁), the stream (4) from the bottom of the column (C2) being sent to the column (C3₁) above the section (S1), and the section (S3) is the single section of a column (C3₂) supplied at its bottom with the stream from the top of the column (C3₁).

7. The method as claimed in claim 6, characterized in that the pressure at the top of the column (C3₁) is 2.7 to 27 kPa, particularly 4 to 15 kPa, and the pressure at the top of the column (C3₂) is 2.7 to 27 kPa, particularly 6.7 to 24 kPa, and in that the temperature at the bottom of each of the columns (C3₁) and (C3₂) is 150 to 250°C, preferably 170 to 210°C, and the temperature at the top of each of the columns (C3₁) and (C3₂) is 40 to 110°C, preferably 60 to 90°C.

8. The method as claimed in claim 1, characterized in that the sections (S1) and (S2) are each formed from at least one evaporator, the stream (4) from the bottom of the column (C2) being sent as feed to the evaporator (E1) or to a first evaporator (E1₁) of a plurality of evaporators mounted in series of the section (S1), the stream (9) containing the absorption solvent or solvents stripped of the lighter compounds being obtained at the bottom of the evaporator (E1) or of the last evaporator (E1₂) of the series (E1₁;E1₂) of the section (S1), and the section (S3) is the single section of a column (C3₃) supplied at its bottom with the stream from the top of the evaporator (E2) or from the last evaporator (E2₂) of a plurality of evaporators mounted in series of the section (S2).

9. The method as claimed in claim 8, characterized in that the pressure at the top of the column (C3₃) is 2.7 to 27 kPa, particularly 6.7 to 24 kPa, and that the temperature at the bottom of column (C3₃) is 150 to 250°C, particularly 170 to 210°C, and the temperature at the top of said column (C3₃) is 40 to 110°C, particularly 60 to 90°C.

10. The method as claimed in one of claims 1 to 9, characterized in that the (meth)acrylic acid concentration in the feed to the section (S1) is 5 to 70% by weight, particularly 10 to 30% by weight.

11. The method as claimed in one of claims 1 to 10, characterized in that the stream (5) of heavy intermediate compounds from the bottom of the section (S3) is sent to a column (C4) adapted for removing, at the top, at least part (8) of the heavy intermediate compounds, and for recovering, at the bottom, a stream (7) of the heavy solvent or solvents and of the polymerization inhibitor or inhibitors initially present in the stream (5) fed to the column (C4), said stream (7) being advantageously recycled as a stabilizing stream at the top of the preceding columns or sections (C1; C2; C3; C3₁; C3₂, C3₃).

12. The method as claimed in claim 11, characterized in that the pressure at the top of the column (C4) is 2.7 to 40 kPa, particularly 9.3 to 20 kPa.

13. The method as claimed in one of claims 1 to 9, characterized in that the stream (9) from the bottom of the section (S1) is recycled to the top of the absorption column (C1), if necessary after removing a stream (11) of heavy impurities having a boiling point higher than that of the solvent or higher than that of the solvent having the highest boiling point.

14. The method as claimed in one of claims 11 to 13, characterized in that, to offset the losses occurring during the purification sequence, fresh solvent or solvents are introduced into the solvent rich loops, particularly into the stream of the bottom

of the section (S1) and of the column (C4) (respectively 13 and 7), recycled at the top of the column (C1), and/or, in the case in which this solvent contains light impurities with a boiling point close to that of the (meth)acrylic acid, additional solvent is fed to the stream (5) feeding the column (C4).

15. The method as claimed in one of claims 11 to 14, characterized in that one or more heavy hydrophobic absorption solvents is used, having a boiling point above 200°C at atmospheric pressure.

16. The method as claimed in claim 15, characterized in that ditolyether is used as a heavy hydrophobic solvent.

17. The method as claimed in one of claims 1 to 16, characterized in that the polymerization inhibitor or inhibitors in the presence of which the absorption is carried out in the column (C1) and the separations are carried in the column (C2) and the sections (S1) to (S3) are selected from phenolic compounds, such as hydroquinone or methylether of hydroquinone, quinones such as benzoquinone, phenothiazine and its derivatives such as methylene blue, manganese salts, such as manganese acetate, metal thiocarbamates such as the copper salts of dithiocarbamic acid, such as copper dibutyldithiocarbamate, the N-oxyl compounds, among which 4-hydroxy-2,2,6,6-tetramethylpiperidinoxyl, amine compounds, such as derivatives of paraphenylene diamine, compounds with a nitroso group such as N-nitrosophenyl hydroxylamine, and ammonium salts of N-nitrosophenyl hydroxylamine.